

Performance of nanotube-based electrodes from temperature-controlled electrophoretic deposition

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Abstract Single-walled carbon nanotube-coated electrodes were fabricated from functionalized nanotube aqueous suspensions by the electrophoretic deposition (EPD) method for a potential large-scale production of nanostructured electrical components. The dependences of nanotube coatings and their electrochemical properties on the processing temperature of EPD were investigated. Optical and scanning electron microscope images were used to examine the microstructure, surface roughness, and thickness of the nanotube-based electrodes. The BET-specific surface area and the average pore size were analyzed. The prepared electrodes were characterized by cyclic voltammetry and their specific capacitances were measured. Correlations between the processing temperature and the electrode capacitance, nanotube loading, specific surface area, and average pore size were experimentally characterized.

Keywords Single walled carbon nanotubes · Network · Electrophoretic · Temperature controlled · Pore size · Cyclic voltammetry

1 Introduction

The modern era of portable electronics and electric vehicles is putting increasing demands on advanced energy conversion and storage devices. In order to reduce the energy dependence on fossil fuels, electronic energy storage devices with larger storage capacities, higher charge rates, and longer operational lifetimes are needed. New and innovative materials and processes are being developed with an effort to increase the energy storage capacities and improve the charging/discharging performance of batteries and capacitors [1, 2]. Electrochemical double-layer capacitors do not rely on chemical reactions for charge transfer and storage. Therefore, they are not limited in the reaction rate or lifetime of chemical kinetics and the charges are created by the ion transportation in an electrolyte and between two opposite electrodes [3]. Using the electric double-layer effect to store charges at the surface of the electrodes, the amount of charges is primarily dependent on the effective surface area available for ion attraction. However, recent studies have shown that the pore size of porous electrode materials can play an important role in determining the amount of charges stored [4]. This role can be attributed to the need of ion transportation channels through pores before they reach the available surface sites. Nanostructured porous electrodes have found their applications as well in other energy conversion and storage devices, such as fuel cells [5, 6], solar cells [7, 8] and batteries [9, 10], etc., where the specific surface area and porosity are major performance indicators.

The literature shows that there is a critical pore size less than 1 nm in diameter which can provide the largest charge storage capability on electrodes. Typical porous materials such as Nickel or Carbon foams and fabrics have pore sizes much larger than this critical value and in the order of

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micrometers [11]. Controlling the pore size is hence critical to optimize the ion transport channel and subsequently increase the amount of stored charges on those electrodes. The use of dispersed single-walled carbon nanotubes (SWCNTs) to form nanopores for facilitating electrolyte ion transportation and trapping more charges could enable tailoring of electronic structures for an optimum storage capacity [12]. A process is therefore needed, which should be repeatable and can uniformly deposit as-produced SWCNTs over a large area, for a high volume production of CNT-based electrodes while producing desired nanopores to create the maximum charge capacity.

In literature various SWCNT and multi-walled CNT coatings, as formed through dispersion of nanotube suspensions by means of drop coating [13, 14], high voltage electro-spraying (HVES) [15], and electrophoretic deposition (EPD) methods [16–18], and their effects on the performance of electronic components were investigated. Among these fabrication methods, the EPD process has been developed and detailed recently [19–21] and its application in fabricating micro-capacitors was highlighted [22]. It was determined in a recent work [15] that the use of EPD with acid-functionalized SWCNTs is an effective method for the creation of electrically conductive nanoporous networks. And, it was found that at room temperature, the linear capacitance gains with the processing time could be achieved within the first 10 min. And, beyond such period, the capacitance gains tapered off following a logarithmic approximation. In order to further characterize the controlling parameters of the EPD process and potentially to provide some guidance for a large-scale fabrication of nanotube-based electrodes by this method, the current study focuses on investigating the influence of its processing temperature on the microstructures and electrochemical properties of the SWCNT coatings. It is worthwhile to note that as an important parameter for industrial manufacturing processes, the influence of EPD processing temperature on the derived coatings and their structural and electrical properties such as specific capacitance has rarely been reported. For this purpose, a new EPD cell was fabricated with the ability to control the temperature of the cell at constant temperatures between 5 and 60 °C. The design of the new EPD cell addressed the possible scale-up issues using an external thermoelectric heater and a simple circuit consisting of a DC power supply and two electrodes. These electrodes can be easily replaced by a roll-up stainless steel sheet and a counter electrode in the industrial scale reel to reel manufacturing [23]. A series of systematic studies was performed for characterizing the correlations between the processing temperature and electrode capacitance, nanotube coating thickness, surface roughness, network average pore size, and specific surface area. The characterization of these correlations allows for

optimizing the EPD process for large-scale manufacturing of SWCNT-based electrodes. The extended EPD processing of SWCNTs was conducted at the controlled temperature to look for a capacitance limit up to 60 min (beyond previously reported 10 min).

2 Experimental

Purified SWCNTs (97 %) obtained from Nano-C Inc. were first acid functionalized using a mixture of concentrated nitric (HNO_3) and sulfuric (H_2SO_4) acid with a ratio of 1:3 respectively (Sigma-Aldrich). 10 mL of the acid mixture was then added to 250 mg of SWCNT powder for 30 min to complete the carboxyl functionalization [24, 25]. During this process the acid mixture added $-\text{COOH}$ functional groups to defect and end sites on the SWCNTs [26, 27]. This treatment negatively polarizes the SWCNTs and allows them to disperse well in pure H_2O without the use of additional surfactants [28]. After acid functionalizing, the SWCNT acid mixture was diluted with de-ionized water and washed through a filter to remove the residual acidic solution from the SWCNTs. Once the SWCNTs were neutralized, the functionalized SWCNTs were washed from the filter into 500 mL of de-ionized water to produce a SWCNT aqueous suspension with a SWCNT concentration of approximately $0.5 \text{ mg mL}^{-1} \text{ H}_2\text{O}$. The suspension was then placed in a Branson 5210 Ultrasonic cleaner for 30 min at 22 °C. Before EPD the suspension was placed in the ultrasonic bath for an additional 5 min and centrifuged for 30 min at 4,000 RPM to remove agglomerated SWCNT bundles from the suspension.

A temperature controlled EPD cell was fabricated with a working distance of 1 cm between electrodes (Fig. 1). 5 mL of functionalized SWCNTs suspension having an SWCNT content of approximately 2.5 mg was used for each process. During fabrication the positive electrode from the EPD cell also acted as the current collector for the SWCNT electrode. Stainless steel (SST) (70 % Fe, 19 % Cr, 11 % Ni: wt%) sheets 0.1 mm thick from Alfa Aesar were used as the current collectors and substrates for the SWCNT electrodes. The SST electrodes were cut, numbered, and weighed before deposition. Then they were installed into the EPD cell and lowered into the functionalized SWCNT aqueous suspension to a depth of 1 cm. A DC power supply was used to apply 40 V to the EPD cell for duration of 3–60 min. A thermoelectric cooler (TEC) was used in the EPD cell to cool or heat the SWCNT suspension and maintained the processing temperature at 5, 22, 45, and 60 °C throughout the EPD process. During EPD processing the negatively charged functionalized SWCNTs moved toward the positive electrode and were uniformly coated the surface. The SWCNT coated

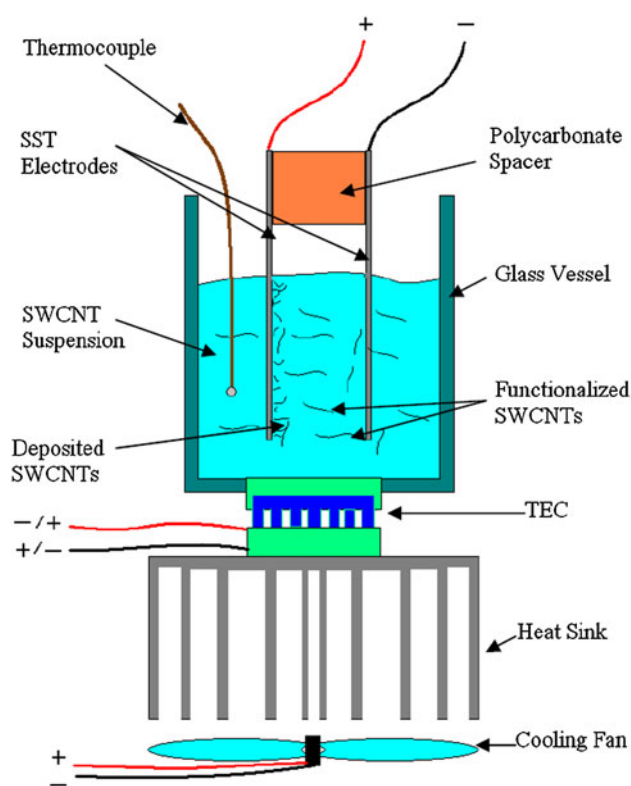


Fig. 1 Schematic diagram of the temperature controlled electrophoretic deposition cell

electrode was removed from the suspension under the applied voltage and dried in an oven at 80 °C for 30 min to remove moisture and to solidify the SWCNT network.

Field emission scanning electron microscope (FE-SEM) images were gathered on a LEO 1530 FE-SEM and were used to visualize the microporosity and evaluate the uniformity of the coated SWCNT networks. For characterizing the specific surface area and average pore size, nitrogen adsorption measurements were performed at 77 K in a Micrometric Floworb surface area analyzer. The as-produced electrodes with the EPD-coated SWCNT network on the SST substrate were used. The geometric area of the SST substrate is 1×1 cm. From the nitrogen adsorption data, the surface area was determined by the Brunauer-Emmett-Teller (BET-N₂) theory. The specific surface area was calculated by deducting the mass and geometric area (1 cm^2) of the SST substrate. The pore sizes of these SWCNT electrodes were determined by means of the Barrett-Joyner-Halenda (BJH) method and Horvath-Kawazoe (HK) technique. Cyclic voltammetry (CV) measurements were performed on a Gamry Reference 3000 Potentiostat within a potential range or -0.2 to 0.8 V. The scan rates were set to 20, 100, and 500 mV s^{-1} , and the measurement were conducted for the SWCNT electrodes produced using EPD processing temperatures of 5,

22, 45, and 60 °C and with different processing times of 3, 8, and 60 min.

3 Results and discussion

Changing the EPD processing temperature had a dramatic effect on the visual quality of the SWCNT coated electrodes. Electrodes processed at higher temperatures showed noticeably darker coatings with a better uniformity. Fewer transparent sections were found on high temperature processed electrodes. In Fig. 2, these samples were fabricated with two EPD processing temperatures, 5 and 60 °C, respectively, with the processing time ranging from 3 to 60 min. For the same processing time, a higher temperature produced a darker SWCNT film. For the same processing temperature, a longer processing time produced a darker SWCNT film. The loading of SWCNTs on the SST substrate was further analyzed quantitatively using SEM images and the mass measurement, as shown later.

Figure 3 shows FE-SEM images of SWCNT-coated electrodes produced using the different processing temperatures. Here, all images refer to the surface view of as-produced electrodes. The processing time was 8 min and the processing temperatures were 5, 22, 45, and 60 °C. These images demonstrate that an increase in the EPD processing temperature resulted in coarser SWCNT networks with larger three dimensional microstructures. As shown in Fig. 3a, the electrode produced at 5 °C exhibits less microstructures and the surface is smoothest. When the processing temperature was 60 °C, on the other hand, as shown in Fig. 3d, more SWCNTs were deposited on the electrode, which caused the formation of more three dimensional microstructures and a rough surface.

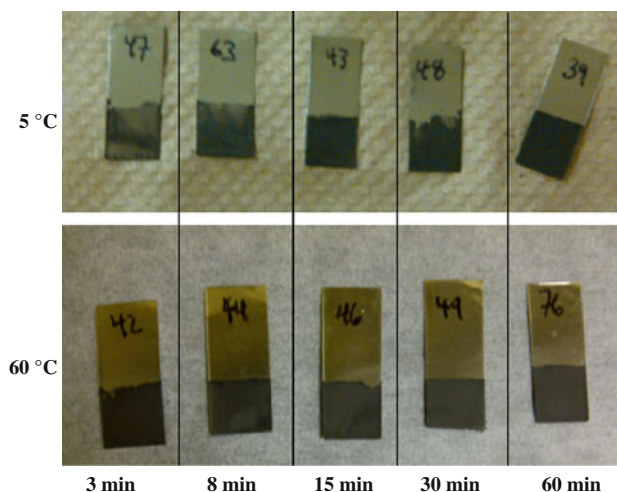


Fig. 2 Optical images of SWCNT-coated electrodes processed at 5 and 60 °C for the processing times of 3–60 min

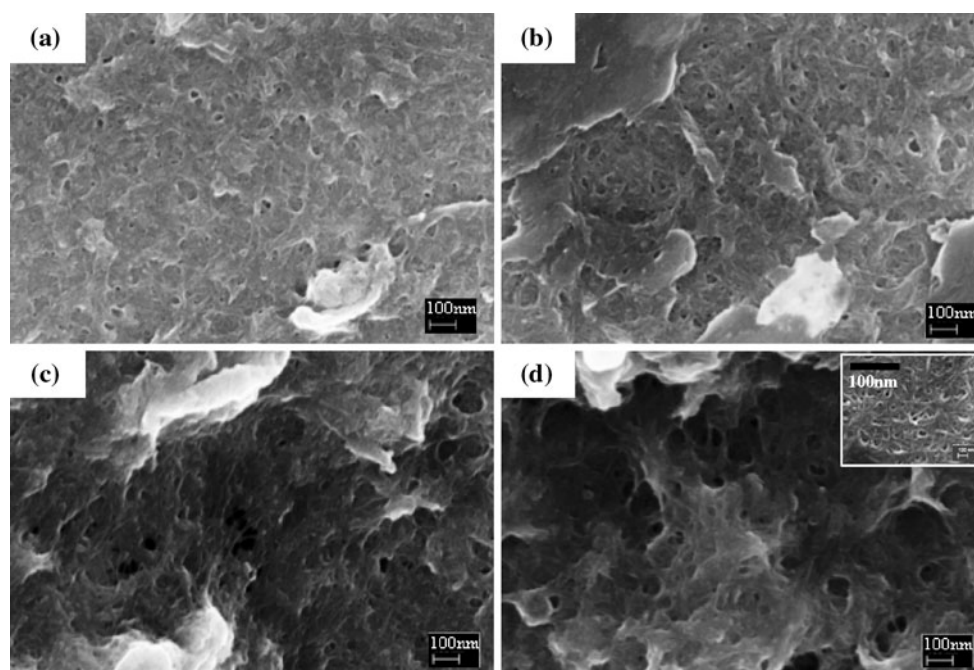


Fig. 3 FE-SEM images of the surface views of SWCNT networks produced using 40 V, with 8 min, and at the different processing temperatures: **a** 5 °C, **b** 22 °C, **c** 45 °C, and **d** 60 °C. The insert in **d** shows an image of the pores formed by carbon nanotubes

The insert in Fig. 3d illustrates the pore structures which were formed in the SWCNT network. The mass of SWCNT films on the as-produced electrodes were measured through comparing the mass before and after EPD processing. It was found that the mass of the SWCNT film changed from 0.3 to 0.6 mg when the processing temperature was increased from 5 to 60 °C (Fig. 4). For a fixed processing time such as 8 min, the mass of SWCNTs on the SST substrate changed from 0.4 to 0.58 mg when the processing temperature increased from 5 to 60 °C.

BET surface area analysis was also performed on SWCNT-coated electrodes processed at 5, 22, 45, and 60 °C for a duration of 3 min, to further investigate the effects of processing temperature on the specific surface area and pore size of the as-produced SWCNT films. Note

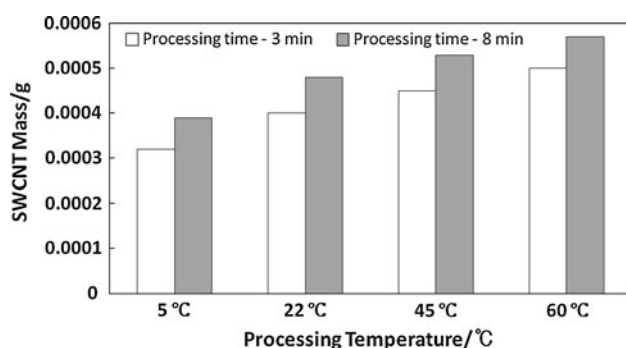


Fig. 4 The mass measurement of as-produced SWCNT films for 3 and 8 min at different processing temperatures

that to characterize the dependence of the specific surface area and average pore size on the EPD processing temperature, a shorter processing time of 3 min was chosen. This helped to eliminate the influence from these aforementioned three dimensional microstructures on the surface. Figure 5 demonstrates that the specific surface area of the SWCNT network increased from 180 to 382 m² g⁻¹ when the EPD processing temperature was changed from 5 to 60 °C, respectively, given the same processing time. This range of specific surface areas of the SWCNT network, which is lower than the reported value of 600 m² g⁻¹ for intrinsic SWCNTs in literature [29], agrees with the reported value (280 m² g⁻¹) for high-power capacitance electrodes fabricated with a composite of SWCNTs and carbon nanohorns [30]. The as-produced SWCNT films in this study contained some agglomerated CNTs which reduced the specific surface area of intrinsic CNTs. The specific surface area of as-produced electrodes changes with the EPD processing time as well [15]. Figure 5 confirms that, similar to the influence of the processing time on the specific surface area, a higher EPD processing temperature would bring more SWCNTs onto the SST substrate, which subsequently yields a greater specific surface area of the SWCNT network.

In addition to the specific surface area, another important structural property influencing the capacitance of the as-produced electrodes is the average pore size of the SWCNT network. It was found that the average pore size was reduced with increasing processing temperatures as

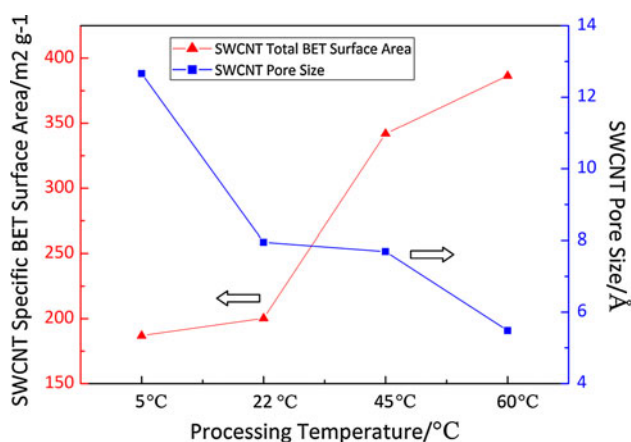


Fig. 5 The BET specific surface area and average pore size measured for electrodes fabricated with various EPD processing temperatures. The processing time is 3 min

roughly shown in Fig. 3 where more visible pores exist for the 5 °C sample. Figure 5 also plots the measured average pore sizes for SWCNT-coated electrodes produced from different processing temperatures. It shows that when the processing temperature increased the average pore size was reduced from approximately 12.7 Å at 5 °C to 5.5 Å at 60 °C. As reported in literature [4, 31], reducing the average pore size below 1 nm (10 Å) can increase the specific capacitance of the CNT-based electrodes. Also, there was some attempt to improve the specific capacitance through annealing/sintering the SWCNT electrode [32]. Similar to our finding, the heat treatment of fabricated SWCNT electrodes has a positive effect on increasing the specific capacitance through reducing the average pore size. In our study, however, the temperature was controlled in the cell during the EPD processing rather than through annealing the produced electrode structure. In addition, the formation mechanisms of nanopores are different in two studies. In literature [32], the reduction of pore sizes to near 30–50 Å could result from stress release and the subsequent structure rearrangement from heat treatment. In our study, the formation of much smaller nanopores around 10 Å was caused by the influence of processing temperature on the mobility of charged SWCNTs and the EPD deposition rate (see below). The previous finding can be viewed as a “top-down” effect while our process provides a “bottom-up” control.

The above analyses on the correlations between the structural properties (including the specific surface area, mass of the SWCNT film and average pore size) of the as-produced electrodes and the EPD processing temperature suggest that the specific surface area and mass of the SWCNT film increase with the processing temperature, while the averaged pore size decreases with the increasing processing temperature. It can be expected that during

EPD, a faster deposition rate of charged SWCNTs moving from the solution to the SST substrate will add more mass onto the electrode, which subsequently results in a larger specific surface area and a denser network with the lower porosity. In order to investigate the effect of the EPD processing temperature on the deposition rate of the SWCNTs, according to the literature [33, 34], the deposition rate (ω) is considered as a function of the concentration of charged particles in the solution (C), the electrical field strength (E), the electrophoretic mobility (μ), the area for deposition (A), and an external factor (f), as shown below

$$\omega = \int_{t_2}^{t_1} f \mu E A C \, dt \quad (1)$$

Furthermore, the electrophoretic mobility of charged particles (μ) can be approximated as a function of the zeta potential (ζ), permittivity (ϵ), and viscosity (η), of the solution, as shown in Henry’s equation

$$\mu = \frac{2\epsilon\zeta}{3\eta} \quad (2)$$

Equation 2 clearly shows that the mobility of particles (the charged SWCNTs in this study) is inversely proportional to the viscosity of the fluid which varies with the temperature. In addition, the researchers found [35] the interaction potential of suspending particles in EPD can be expressed as a function of the processing temperature and subsequently affects the deposition rate. In good agreement with these mechanisms, it was observed in this study that for EPD-fabricated SWCNT networks, both the specific surface area and average pore size of the deposited films are significantly affected by the processing temperature. In summary, the processing temperature affects the mobility of charged particles (SWCNTs) in the suspension. A higher temperature results in a greater mobility. And, the deposition rate of EPD is determined by the mobility of SWCNTs in the suspension. A greater mobility will lead to a larger deposition rate. When the processing temperature increases, the mobility of charged SWCNTs in the suspension increases and a large deposition rate is achieved. With the increasing temperature, more SWCNTs are deposited on the SST substrate, which further increases the specific surface area, reduce the pore size, and increase the mass of the film.

The influence of the processing temperature on the amount of SWCNTs added to the electrode was further examined with the cross-sectional analysis of the as-produced film. This was done on the SWCNT coatings by mounting the samples in an epoxy resin and polishing perpendicular to the coating direction. FE-SEM images of

the cross-sectioned samples were then gathered to determine the thickness and surface roughness of the SWCNT coatings produced under the different EPD processing temperatures and at the same processing time. From the cross-sectional images, the SWCNT coating thicknesses were determined to be: 4.5–5.3 μm for 5 $^{\circ}\text{C}$, 2.9–6.6 μm for 22 $^{\circ}\text{C}$, 7.5–18.8 μm for 45 $^{\circ}\text{C}$, and 9.9–20.1 μm for 60 $^{\circ}\text{C}$, with all processed for 8 min durations. An example of these cross-sectional FE-SEM images from the 60 $^{\circ}\text{C}$ sample is shown in Fig. 6.

From the cross-sectional data, the thickness and surface roughness variation as a function of the processing temperature were plotted. In Fig. 7, the range of the measured SWCNT film thicknesses from different locations on the sample, which was fabricated at one of four processing temperatures, is plotted and the average value is indicated. Although there does not appear to be a direct linear correlation between the average thickness and the processing temperature, the trend of increasing roughness and thickness with increasing processing temperature is apparent for higher temperatures. This agrees well with the BET data showing increasing specific surface area with increasing EPD processing temperatures (Fig. 5).

To determine the effect of the EPD processing temperature on the specific capacitance of the SWCNT electrodes, CV curves were recorded for each processed electrode using an Ag/AgCl reference electrode and a Pt counter electrode in a three-electrode cell. The working electrolyte for the CV curves was 1 M H_2SO_4 . From the CV plots shown in Fig. 8a, the specific capacitance of the electrodes can be characterized by integrating the areas under the curves [36, 37]. Figure 8b shows the derived specific capacitance values of EPD-produced electrodes for different processing times of 0–8 min and with the processing

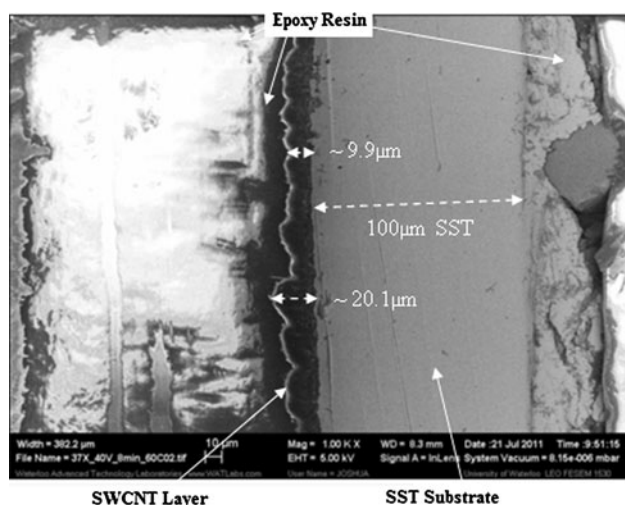


Fig. 6 The FE-SEM image of the cross section of a SWCNT-coated SST electrode produced using 40 V, with 8 min and at 60 $^{\circ}\text{C}$

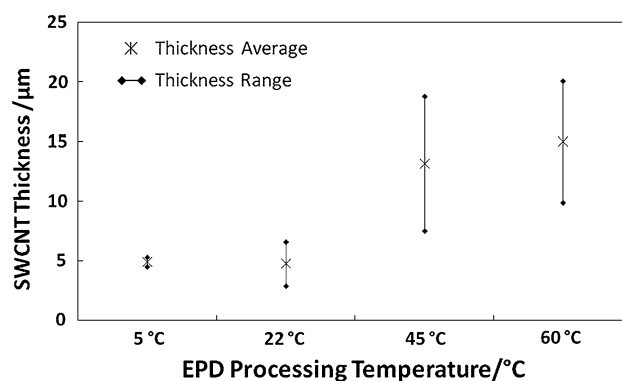


Fig. 7 The SWCNT film thickness and surface roughness are plotted as a function of the EPD processing temperature for the as-produced electrodes with 8 min processing

temperatures from 5 to 60 $^{\circ}\text{C}$. The scan rate was set to 20 mV s^{-1} .

From Fig. 8b a, direct correlation between the specific capacitance and EPD processing temperature can be observed. The slope of the specific capacitance curve for the processing temperature of 60 $^{\circ}\text{C}$ is almost three times steeper than the slope for the 5 $^{\circ}\text{C}$ curve. Likewise, the specific capacitance of the electrode processed at 60 $^{\circ}\text{C}$ for 8 min is 2.8 times larger than the electrode processed at

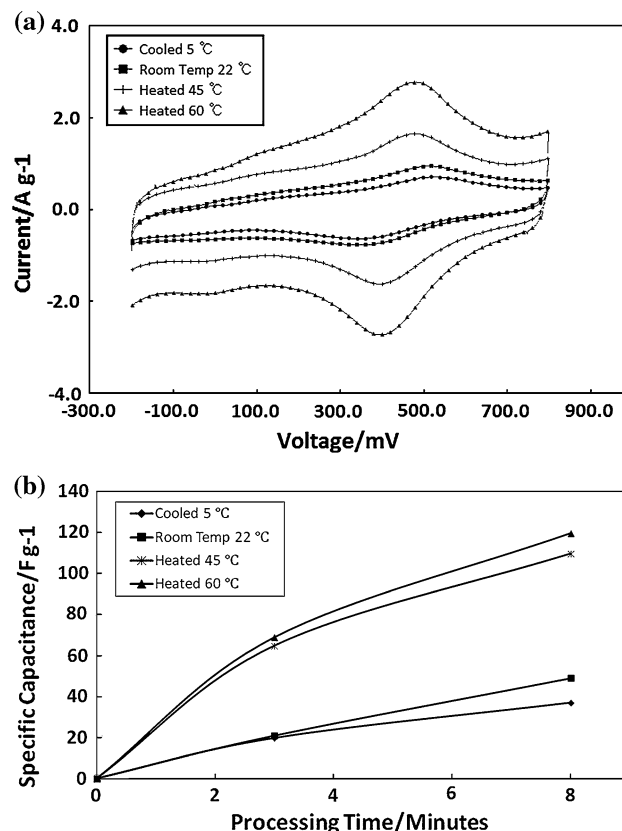


Fig. 8 a Cyclic voltammograms of the as-produced electrodes with 8 min processing in 1 M H_2SO_4 ; **b** The derived specific capacitances

5 °C for the same processing time. These values are 119 and 42 F g⁻¹, respectively. In addition, increasing the processing temperature can reduce the EPD time for producing the desired specific capacitance. As shown in the figure, increasing the processing temperature from the room temperature to 60 °C has reduced the processing time by 25 %. This information is crucial for the optimization of processing parameters for the fabrication of SWCNT-coated electrodes in high volume manufacturing.

Figure 9 shows the relationship between the specific capacitance of the SWCNT-coated electrode and the SWCNT film thicknesses, while Fig. 10 shows the relationship between the specific capacitance and the specific surface area. Note that it was very difficult to accurately measure the thickness of the SWCNT film due to the complex cross-sectional morphology shown in Fig. 6. In Fig. 9, the range of this thickness of the SWCNT film was measured for each sample and these ranges were plot with their average values. The EPD process times were 8 min for all these samples. From these plots in Figs. 9, 10, a linear dependence could be observed for both relationships. In both cases, an increase in the EPD processing temperature results in an increase in the specific capacitance which is linearly dependent on the thickness and specific surface area of the SWCNT film. This observation, made for the different processing times of 3 min and 8 min, is an important finding. It demonstrates that regardless of the SWCNT film thickness, as long as the average pore size is around 1 nm, electrolyte ions can penetrate through the full thickness of the SWCNT film and access to the larger surface area of the EPD-produced SWCNT network. In other words, the relationship between the specific capacitance and the specific surface area becomes linear when the pore sizes are distributed in a relative uniform way and in an optimized range (around 1 nm as shown in literature [4]). The specific capacitance measured on the electrode which was fabricated at 60 °C and with 8 min is 119 F g⁻¹.

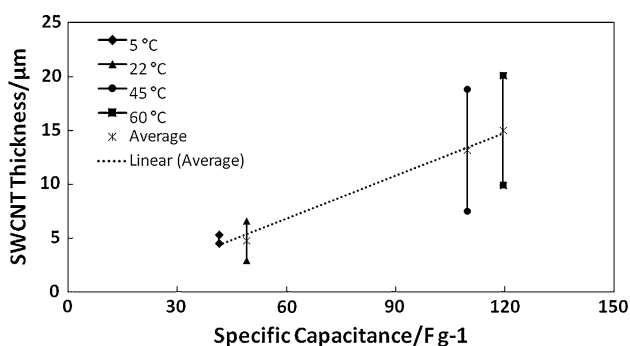


Fig. 9 The linear dependence of the specific capacitance of the electrodes on the SWCNT film thickness. The processing time was 8 min

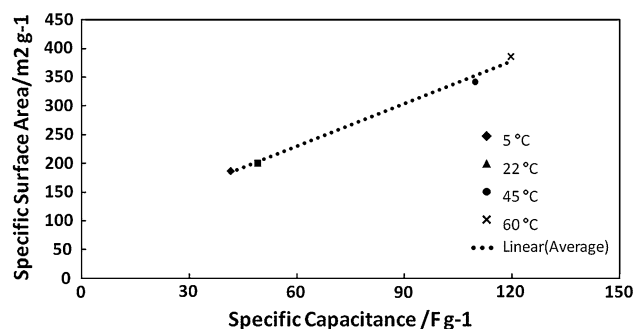


Fig. 10 The linear dependence of the specific capacitance of the electrodes on the specific surface area of the SWCNT network. The processing time was 3 min

This value is close to the specific capacitance reported previously for multi-walled CNT-based electrodes (113 F g⁻¹) [38].

4 Conclusion

The bulk SWCNT powders were used to produce CNT-coated electrodes using a temperature-controlled EPD process and the effects of EPD processing temperature on the structural properties and specific capacitance of as-produced electrodes were studied. A custom EPD cell was fabricated which allowed for the deposition of SWCNTs onto the surface of stainless steel current collectors at processing temperatures between 5 and 60 °C. The loading of the SWCNTs on the electrodes were analyzed and measured using the optical images, mass measurement, and FE-SEM images. The mass of added SWCNTs on the SST substrate changed with the processing temperature and time of the EPD process. When the processing time increases, more SWCNTs were added onto the SST substrate. For a higher temperature, more mass was deposited. In addition, BET surface area analysis and SEM images of cross-sectioned samples showed that increasing the EPD-processing temperature resulted in increasing the SWCNT network thickness, surface roughness, and specific surface area. The EPD process yielded SWCNT-coated electrodes with nanopores of about 1 nm and the characterizations showed that increasing the EPD processing temperature reduced the average pore size from 1.3 nm at 5 °C to 0.6 nm at 60 °C. A linear correlation was observed between the specific capacitance of the electrode and the specific surface area of the SWCNT film, which suggests electrolyte ions have a full access to available surface area sites throughout the SWCNT network. The correlations and process optimizations identified in this study will help develop the cost-effective fabrication technology for producing SWCNT-coated electrodes.

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